

(19)



Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number:

0 322 027
A2

(12)

EUROPEAN PATENT APPLICATION

(21) Application number: 88202854.1

(51) Int. Cl. 4: **A23D 3/00 , A23D 5/00 ,**
A23G 1/00 , A23G 9/02

(22) Date of filing: 13.12.88

(30) Priority: 15.12.87 US 132400

(43) Date of publication of application:
28.06.89 Bulletin 89/26

(84) Designated Contracting States:
AT BE CH DE FR GB GR IT LI LU NL SE

(71) Applicant: **THE PROCTER & GAMBLE**
COMPANY
One Procter & Gamble Plaza
Cincinnati Ohio 45202(US)

(72) Inventor: **Seiden, Paul**
2890 Grandin Road
Cincinnati Ohio 45208(US)

(74) Representative: **Suslic, Lydia et al**
Procter & Gamble European Technical
Center N.V. Temselaan 100
B-1820 Strombeek-Bever(BE)

(54) **Reduced calorie fats made from triglycerides containing medium and long chain fatty acids.**

(57) A reduced calorie fat comprising at least about 15% by weight triglycerides selected from the group consisting of MML, MLM, LLM, and LML triglycerides, and mixtures thereof; wherein M = fatty acids selected from the group consisting of C₆ to C₁₀ saturated fatty acids, and mixtures thereof, and L = fatty acids selected from the group consisting of C₁₇ to C₂₆ saturated fatty acids, and mixtures thereof is disclosed. The fat also has the following fatty acid composition: (a) from about 15% to about 70% C₆ to C₁₀ saturated fatty acids; (b) from about 10% to about 70% C₁₇ to C₂₆ saturated fatty acids; (c) not more than about 10% fatty acids selected from the group consisting of C_{12:0} and C_{14:0}, and mixtures thereof; (d) not more than about 20% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, C_{18:3}, and mixtures thereof; and (e) not more than 4% C_{18:2} fatty acids.

EP 0 322 027 A2

REDUCED CALORIE FATS MADE FROM TRIGLYCERIDES CONTAINING MEDIUM AND LONG CHAIN FATTY ACIDS

Cross Reference to Related Application

This application is a continuation-in-part of U.S. application Serial No. 132,400, filed December 15, 1987.

Technical Field

The present invention relates to the field of reduced calorie fats, in particular fats made from triglycerides containing combinations of medium and long chain fatty acids.

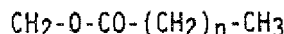
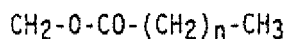
Background of the Invention

Typical vegetable oils and animal fats used in foods contain fatty acids which are predominantly 16 or 18 carbons long and contain zero to three double bonds. These are generally referred to as long chain triglycerides. Some oils such as rapeseed oil contain fatty acids having 20 or 22 carbons or higher.

Medium chain triglycerides (MCT'S) are triglycerides made with saturated C_6 to C_{12} fatty acids. These shorter chain triglycerides are metabolized differently from long chain triglycerides by the body because they are more water-soluble than long chain triglycerides. Because they hydrolyze rapidly and are absorbed via the portal vein, they provide a source of quick energy.

Several references disclose triglycerides containing medium chain and long chain fatty acids. For example, U.S. Patent 3,353,964 of Seiden, issued November 21, 1967, discloses a margarine oil made from corandomized triglycerides containing saturated short chain fatty acids having 6-14 carbon atoms and saturated long chain fatty acids having 20-22 carbon atoms. The triglycerides, a corandomized blend of hydrogenated rapeseed oil with coconut and/or palm kernel oil, are high in lauric acid.

U.S. Patent 4,607,052 of Mendy et al., issued August 19, 1986, discloses triglycerides of the formula:



where R represents an acyl fragment of a polyunsaturated fatty acid containing 18 to 22 carbon atoms, the acyl fragment being capable of being oxidized, and where n represents an integer varying from 2 to 16. The triglycerides are used as nutritional supplements to provide a source of polyunsaturated fatty acids.

A synthetic therapeutic oil is disclosed in U.S. Patent 3,450,819 of Babayan et al., issued June 17, 1969. The oil is useful for treating humans suffering from malabsorption of fat. The oil comprises triglycerides having a major portion of medium chain (saturated C_6 to C_{12}) fatty acids, and a minor portion of essential fatty acids. The essential fatty acids are unsaturated fatty acids, primarily linoleic ($C_{18:2}$), linolenic ($C_{18:3}$), and arachidonic ($C_{20:4}$).

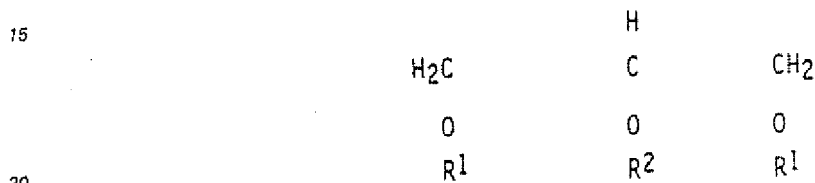
U.S. Patent 4,528,197 of Blackburn, issued July 9, 1985, discloses a composition for enhancing protein anabolism in an hypercatabolic mammal. The composition is made of a nutritionally sufficient source of amino acids, carbohydrates and lipids, the lipids comprising a controlled triglyceride source which, on hydrolysis, yields both long chain fatty acids and medium chain fatty acids. One such fatty acid source disclosed is a structured lipid containing medium chain fatty acids (saturated C_6 , C_{10} , and C_{12}), and essential fatty acids.

Mok et al., "Structured Medium Chain and Long Chain Triglyceride Emulsions are Superior to Physical Mixtures in Sparing Body Protein in the Burned Rat", Metabolism, Vol. 33, No. 10, pp. 910-915, Oct. 1984,

describe an emulsion consisting of triglycerides composed of medium chain and long chain fatty acids in similar proportions used for sparing body protein in burned rats. The triglycerides were made from capric acid ($C_{8:0}$), caprylic acid ($C_{10:0}$), linoleic acid ($C_{18:2}$), and other long chain fatty acids.

Maiz et al., "Protein Metabolism During Total Parenteral Nutrition (TPN) in Injured Rats Using Medium Chain Triglycerides," *Metabolism*, Vol. 33, No. 10, pp. 901-909, October 1984, disclose a lipid emulsion made from triglycerides containing 60% medium chain fatty acids and 40% long chain fatty acids said to improve protein utilization in injured rats. The medium chain fatty acids used are saturated C_8 through C_{12} , and the long chain fatty acids are derived from sunflower or safflower oil (which consist primarily of mixed triglycerides of linoleic and oleic fatty acid moieties).

European Patent Application 216,419 of Jandacek et al., published April 1, 1987, relates to a nutritional fat suitable for use in enteral and parental products, consisting essentially of from about 50% to about 100% by weight of triglycerides having the formula:



The R^1 groups consist of saturated medium chain fatty acids with chain lengths of 7-11 carbon atoms, and the R^2 group consists of 0-90% saturated C_7 - C_{18} fatty acids, 0-90% oleic $C_{18:1}$, 10-100% linoleic ($C_{18:2}$), and 0-10% linolenic ($C_{18:3}$).

U. S. Patent 2,874,056 of Drew, issued February 17, 1959, discloses a triglyceride composition useful in margarines. The triglyceride is made from a combination of medium chain fatty acids ($C_{8:0}$ through $C_{12:0}$) and palmitic acid ($C_{16:0}$).

The Captex 810 series of oils (Capital City Products, Dept. TR, P.O. Box 569, Columbus, OH 43216) contains random structure triglycerides that are made from mixtures of various ratios of long and medium chain fatty acids. The fatty acid compositions of these oils are as follows:

| Fatty acid composition (weight %) of the Captex 810 series | | | |
|--|-------------------------|--|-------|
| Captex series | Linoleic ($C_{18:2}$) | Octanoic and Decanoic ($C_{8:0}$ & $C_{10:0}$) | Other |
| 810A | 10 | 80 | 10 |
| 810B | 25 | 60 | 15 |
| 810C | 35 | 46 | 19 |
| 810D | 45 | 32 | 23 |

None of these references discloses or suggests the reduced calorie fats of the present invention, or the benefits associated therewith. For example, the Seiden patent discloses a margarine oil high in lauric acid. The object is improved eating quality and heat resistance, not calorie reduction. Additionally, lauric acid is metabolized differently from the medium chain (saturated C_6 to C_{10}) fatty acids used in the present invention.

Moreover, the references by Mendy et al., Babayan et al., Blackburn, Jandacek et al., etc., relate to fats useful as nutritional supplements. The fats of the present invention, on the other hand, combine a calorie reduction benefit with good taste. The prior references also have fatty acid compositions different from those of the present invention.

It is, therefore, an object of the present invention to provide fat compositions that are reduced in calories when compared to typical chain length triglycerides.

It is another object of the present invention to provide reduced calorie fats that can be used to make food and beverage products having excellent textural and organoleptic properties.

These and other objects of the present invention will become evident from the disclosure herein.

All parts, percentages, and ratios used herein are by weight unless otherwise indicated.

Summary of the Invention

The invention relates to a reduced calorie fat comprising at least about 15% by weight triglycerides selected from the group consisting of MML, MLM, LLM, and LML triglycerides, and mixtures thereof; wherein M = fatty acids selected from the group consisting of C₆ to C₁₀ saturated fatty acids, and mixtures thereof, and L = fatty acids selected from the group consisting of C₁₇ to C₂₆ saturated fatty acids, and mixtures thereof; and wherein the fat has the following fatty acid composition by weight percent:

- (a) from about 15% to about 70% C₆ to C₁₀ saturated fatty acids;
- (b) from about 10% to about 70% C₁₇ to C₂₆ saturated fatty acids;
- (c) not more than about 10% fatty acids selected from the group consisting of C_{12:0} and C_{14:0}, and mixtures thereof;
- (d) not more than about 20% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, C_{18:3}, and mixtures thereof and
- (e) not more than 4% C_{18:2} fatty acids.

The reduced calorie fats have excellent organoleptic properties, and can be used in a wide variety of food products (e.g., salted and/or fried snacks, other snacks, desserts, baking mixes and other prepared mixes, processed meat products, frozen entrees, chocolate-type products, ice cream and other frozen desserts, salad dressings, frying and baking shortenings, salad oils, margarines, spreads, prewhipped toppings, peanut butter, frostings, confectionery fillings and other confectioneries, cookies, cakes, pie crusts, pastry crusts, breads and other baked goods, and other baking, cooking or frying products. The fats can also be used as pharmaceutical carriers.

Detailed Description of the Invention

Triglyceride fats made with long chain or very long chain saturated fatty acids are reduced in calories because the fatty acids are only poorly absorbed and metabolized by the body. However, the high melting point of these fatty acids gives them a waxy, unpalatable taste. For example, tristearin and tribehenin are low calorie fats, but they are seldom used in foodstuffs because of their waxiness. Therefore, there is a need for a means of making these waxy long chain fats into good-tasting fats that are still reduced in calories.

It has now been surprisingly found that fat compositions containing triglycerides made with a particular combination of saturated long chain fatty acids and saturated medium chain fatty acids provide good taste while still providing a reduction in calories. Thus, the present reduced calorie fats give the advantages of long chain triglycerides without their taste disadvantages.

This finding was unexpected for several reasons. When lauric acid is esterified to a saturated long chain triglyceride, the melting point of the triglyceride is lowered to some extent, but the triglyceride still tastes waxy because it does not melt at body temperature. By contrast, it has now been discovered that when medium chain fatty acids are esterified to such a triglyceride, the melting point of the triglyceride is lowered to a surprisingly much greater extent, so that the triglyceride melts below body temperature and does not taste waxy. This property is highly important for making acceptable foods.

Additionally, it was not clear in view of the prior art that when long chain fatty acids are combined with medium chain fatty acids on a triglyceride molecule, that the combination would provide a calorie reduction benefit. A methodology had to be developed to demonstrate a reduction in absorption.

The present invention is a reduced calorie fat comprising at least about 15% by weight reduced calorie triglycerides selected from the group consisting of MML, MLM, LLM, and LML triglycerides, and mixtures thereof; wherein M = fatty acids selected from the group consisting of C₆ to C₁₀ saturated fatty acids, and mixtures thereof, and L = fatty acids selected from the group consisting of C₁₇ to C₂₆ saturated fatty acids, and mixtures thereof; and wherein the fat has the following fatty acid composition by weight percent:

- (a) from about 15% to about 70% C₆ to C₁₀ saturated fatty acids;
- (b) from about 10% to about 70% C₁₇ to C₂₆ saturated fatty acids;
- (c) not more than about 10% fatty acids selected from the group consisting of C_{12:0} and C_{14:0}, and mixtures thereof;
- (d) not more than about 20% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, C_{18:3}, and mixtures thereof; and

(e) not more than 4% C_{18:2} fatty acids.

The key to the present reduced calorie fats is their combination of particular long chain fatty acid moieties with medium chain fatty acid moieties. As discussed above, the medium chain fatty acids lower the melting point of the fats, and different fatty acid combinations can be used to control the fats' physical properties for specific food applications. This results in a good-tasting fat having good mouthmelt, texture and flavor display.

Moreover, because they do not taste waxy, the present fats can be used in a wide variety of food products, and at higher concentration in the products to afford a greater calorie reduction. This is in contrast to the more limited use of a more waxy-tasting triglyceride, e.g., a triglyceride containing lauric acid and long chain fatty acids. This nonwaxy taste benefit is particularly evident in chocolate products made with preferred reduced calorie fats of the present invention. As measured by differential scanning calorimetry (DSC), chocolate products containing these preferred fats are completely melted at a temperature of from 94° to 96° F. Most of the melting of these chocolate products also occurs in the fairly narrow temperature range of from 80 to 94° F.

Another advantage of the present fats is that they contain only limited amounts of saturated C₂ to C₁₆ fatty acids. Ingestion of large amounts of these fatty acids is known to promote hypercholesterolemia.

The present fat compositions also provide some of the benefits of medium chain triglycerides. For example, the medium chain fatty acids are readily hydrolyzed from the triglycerides. These hydrolyzed medium chain fatty acids are absorbed and then transported directly to the liver (via the hepatic portal vein) where they are extensively oxidized to provide a rapid energy source.

The reduced calorie fats can be used as a partial or complete replacement for the fat component in food products, permitting at least about 10% reduction in calories, and preferably at least about 30% reduction in calories over typical chain length triglycerides (i.e., corn oil), and usually between about 20% and 50% reduction in calories.

For the purposes of the present invention, the reduction in calories provided by the present reduced calorie fats is based on the net energy gain (in Kcal) of rats that have ingested a diet containing the reduced calorie fats, relative to the net energy gain (in Kcal) of rats that have ingested an identical diet, but containing corn oil instead of the reduced calorie fat. The test diets used are nutritionally adequate to support both maintenance and growth of the rats. Total food intake and fat/oil intake are also matched between the two diet groups so that differences in net carcass energy gain are due entirely to the utilizable energy content of the fat/oil. "Net energy gain" is based on the total carcass energy (in Kcal) of the rat fed the test diet for some period of time (usually 4 weeks), reduced by the mean starting carcass energy (in Kcal) determined from a study of a different group of rats of the same sex, strain, and similar body weight fed a test diet that does not contain the fat/oil. "Total carcass energy" is determined by the dry carcass energy per gram (Kcal per gram) multiplied by the dry weight of the carcass (in grams). "Carcass energy per gram" is based on the carcass energy (in Kcal) as determined by bomb calorimetry of a homogeneous sample of the total dry carcass. All of these energy values are the average of a representative sample of rats (i.e., 10 rats).

By "medium chain fatty acids," as used herein, is meant C_{6:0} (caproic), C_{8:0} (caprylic), or C_{10:0} (capric) fatty acids, or mixtures thereof. The C₇ and C₉ saturated fatty acids are not commonly found, but they are not excluded from the possible medium chain fatty acids. The present medium chain fatty acids do not include lauric acid (C_{12:0}), sometimes referred to in the art as a medium chain fatty acid.

By "long chain fatty acids," as used herein, is meant C_{17:0} (margaric), C_{18:0} (stearic), C_{19:0} (nonadecylic), C_{20:0} (arachidic), C_{21:0} (heneicosanoic), C_{22:0} (behenic), C_{23:0} (tricosanoic), C_{24:0} (lignoceric), C_{25:0} (pentacosanoic), or C_{26:0} (cerotic) fatty acids, or mixtures thereof.

In the above listing of fatty acid moieties, the common name of the fatty acid is given following its C_{x:y} designation (wherein x is the number of carbon atoms, and y is the number of double bonds).

By "MML," as used herein, is meant a triglyceride containing a long chain fatty acid in the #1 or #3 position (an end position) with two medium chain fatty acids in the remaining two positions. The absorption of long chain fatty acids is generally reduced in the end positions. Similarly, "MLM" represents a triglyceride with a long chain fatty acid in the #2 position (the middle position) and two medium chain fatty acids in the #1 and #3 positions. "LLM" represents a triglyceride with a medium chain fatty acid in the #1 or #3 position and two long chain fatty acids in the remaining two positions, and "LML" represents a triglyceride with a medium chain fatty acid in the #2 position and two long chain fatty acids in the #1 and #3 positions.

By "stearic MCT," as used herein, is meant a mixture of triglycerides according to the present invention that have been prepared by combining predominantly stearic acid (C_{18:0}) and medium chain fatty acids in

some manner, for example by random rearrangement of tristearin and medium chain triglycerides. The stearic MCT will contain predominantly stearic acid as the long chain fatty acid. By "behenic MCT" is meant a mixture of triglycerides that have been prepared by combining predominantly behenic acid ($C_{22:0}$) and medium chain fatty acids, for example by random rearrangement of tribehenin and medium chain triglycerides. By "stearic/behenic MCT" is meant a mixture of triglycerides that have been prepared by combining predominantly stearic acid, behenic acid, and medium chain fatty acids.

The present reduced calorie fat comprising triglycerides having combinations of medium and long chain fatty acids will preferably contain not more than about 5% by weight $C_{6:0}$ fatty acid, and most preferably not more than about 0.5%. It is also preferred that the fat contain not more than about 7% by weight saturated C_{24} to C_{26} fatty acids, and most preferably not more than about 1%. Preferred reduced calorie fats of the present invention comprise from about 30 to about 55% by weight C_8 to C_{10} saturated fatty acids and from about 30 to about 55% by weight C_{18} to C_{22} saturated fatty acids.

Stearic MCT's according to the present invention will preferably have a carbon number profile of at least about 55% C_{34} to C_{38} , and contain at least about 40% by weight C_6 to C_{10} saturated fatty acids and about 35% to about 50% by weight C_{18} saturated fatty acid.

The reduced calorie fat of the present invention can contain limited amounts of other fatty acids besides medium and long chain fatty acids, without losing the benefits of the invention. As indicated above, small amounts of $C_{12:0}$, $C_{14:0}$, $C_{18:1}$, $C_{18:2}$ and $C_{18:3}$ fatty acids can be present.

Palmitic acid ($C_{16:0}$) is about 95% absorbed by the body, while the longer chain fatty acids are less absorbed. Therefore, it is preferred that the present reduced calorie fat contain not more than about 10% by weight $C_{16:0}$ fatty acid.

In another preferred embodiment, the reduced calorie fat will contain not more than about 6% by weight fatty acids selected from the group consisting of $C_{18:1}$, $C_{18:2}$, $C_{18:3}$, and mixtures thereof, more preferably no more than about 1%, and most preferably not more than about 0.5%. Preferred reduced calorie fats also contain not more than about 3%, and more preferably no more than about 1%, by weight fatty acids selected from the group consisting of $C_{12:0}$ (lauric) and $C_{14:0}$ (myristic), and mixtures thereof. Whereas the medium chain fatty acids (C_6 to C_{10}) are absorbed by the body via the portal vein, lauric and myristic are absorbed via the lymphatic system. Lauric and myristic also result in more fat deposition than medium chain fatty acids.

For optimum taste and calorie reduction, it is also preferred that the reduced calorie fats of the present invention comprise at least about 30% by weight of the triglycerides containing combinations of medium and long chain fatty acids (i.e., MML, MLM, LLM and LML triglycerides), more preferably at least about 50% by weight of these triglycerides, and most preferably at least about 80% by weight of these triglycerides. Preferred reduced calorie fats of the present invention comprise at least about 10% by weight of a mixture of MML and MLM triglycerides, more preferably at least about 35% by weight of such combined triglycerides, and most preferably at least about 70% by weight of such combined triglycerides. Preferred reduced calorie fats also comprise not more than about 90% by weight combined LLM and LML triglycerides, more preferably not more than about 65% by weight LLM and LML triglycerides, and most preferably not more than about 30% by weight combined LLM and LML triglycerides. For most uses, these preferred reduced calorie fats also comprise minimized levels of MMM triglycerides and LLL triglycerides. By "MMM," as used herein, is meant a triglyceride containing medium chain saturated fatty acid residues at all three positions. Similarly, "LLL" represents a triglyceride containing long chain saturated fatty acid residues at all three positions. These preferred reduced calorie fats comprise not more than about 15% by weight, more preferably not more than about 10% by weight, and most preferably not more than about 5% by weight MMM triglycerides. These preferred reduced calorie fats also comprise not more than about 5% by weight, more preferably not more than about 2% by weight, and most preferably not more than about 1% by weight LLL triglycerides. However, for ice creams and ice cream coatings, these reduced calorie fats preferably comprise from about 10 to about 15% by weight MMM triglycerides.

Certain reduced calorie fats of the present invention are particularly preferred for chocolate and other confectionery products. These particularly preferred reduced calorie fats comprise at least about 93% by weight of a mixture of MML and MLM triglycerides, more preferably at least about 96% by weight of such combined triglycerides, and most preferably at least about 97% by weight of such combined triglycerides. These preferred reduced calorie fats also comprise not more than about 7% by weight combined LLM and LML triglycerides, more preferably no more than about 4% by weight LLM and LML triglycerides, and most preferably no more than about 2.5% by weight combined LLM and LML triglycerides. These particularly preferred reduced calorie fats further comprise no more than about 1%, preferably no more than about 0.5% and most preferably no more than about 0.2% by weight MMM triglycerides, and no more than about 2% by weight, preferably no more than about 1% by weight and most preferably no more than about 0.5%

by weight LLL triglycerides.

These preferred fats for chocolate and other confectionery products also have the following preferred and most preferred carbon number profiles (CNP).

| CNP | PREFERRED (%) | MOST PREFERRED (%) |
|--------------|---------------|--------------------|
| 30 or lower | < 0.4 | < 0.2 |
| 34 | < 0.3 | < 0.2 |
| 36 | < 3.5 | < 2.5 |
| 38 | 22-42 | 25-40 |
| 40 | 32-55 | 40-50 |
| 42 | 5-30 | 12-25 |
| 44 | < 1.5 | < 1.2 |
| 46 | < 1 | < 0.6 |
| 48 | < 0.8 | < 0.7 |
| 50 | < 0.6 | < 0.5 |
| 52 | < 0.4 | < 0.3 |
| 54 or higher | < 0.9 | < 0.4 |

Table 1 below illustrates some of the possible triglyceride variations within the MML/M~~M~~M and LLM/LML groups. Combinations of different medium and long-chain fatty acids on the triglycerides are correlated with the carbon numbers of the triglycerides. (The list is not meant to be exhaustive.) The table shows that a wide variety of triglycerides exist at a given carbon number (CNP).

Table 1

Some of the Possible Triglycerides of Medium*
and Saturated Long Chain** Fatty Acids

5

| | <u>CNP</u> | | | <u>MLM & LMM</u> | | |
|----|------------|----------|----------|----------------------|---------|----------|
| | 34 | 8-8-18 | 6-8-20 | 6-6-22 | 8-10-16 | |
| | | 8-18-8 | 6-20-8 | 6-22-6 | 8-16-10 | |
| 10 | | | 8-6-20 | | 10-8-16 | |
| | 36 | 8-10-18 | 8-8-20 | 6-10-20 | 6-8-22 | 6-6-24 |
| | | 8-18-10 | 8-20-8 | 6-20-10 | 6-22-8 | 6-24-6 |
| | | 10-8-18 | | 10-6-20 | 8-6-22 | 10-10-16 |
| 15 | | | | | | 10-16-10 |
| | 38 | 10-10-18 | 8-10-20 | 8-8-22 | 8-6-24 | 6-10-22 |
| | | 10-18-10 | 8-20-10 | 8-22-8 | 8-24-6 | 6-12-20 |
| | | | 10-8-20 | | 6-22-10 | 12-6-20 |
| | | | | | 6-8-24 | 10-6-22 |
| 20 | 40 | 8-10-22 | 10-10-20 | 8-8-24 | 6-10-24 | |
| | | 8-22-10 | 10-20-10 | 8-24-8 | 6-24-10 | |
| | | 10-8-22 | | | 10-6-24 | |
| | 42 | 10-10-22 | 8-10-24 | | | |
| 25 | | 10-22-10 | 8-24-10 | | | |
| | | | 10-8-24 | | | |
| | 44 | 10-10-24 | | | | |
| 30 | | 10-24-10 | | | | |

30

*Saturated medium chain fatty acids (M) chain length:
6, 8, 10.

**Saturated long chain fatty acids (L) chain length:
16, 18, 20, 22, 24. (While palmitic acid (C₁₆) is
35 included here as a long chain fatty acid for illustra-
tion purposes, it is not within the claim definition
of a long chain fatty acid.)

40

45

50

55

LLM & LML

| | | | | | |
|----|----|----------------------------------|----------------------------------|----------------------------------|----------------------|
| 5 | 38 | 6-16-16 16-6-16 | | | |
| | 40 | 6-16-18 6-18-16 16-6-18 | 8-16-16 16-8-16 | | |
| 10 | 42 | 6-18-18 18-6-18 | 8-16-18 8-18-16 16-8-16 | | |
| 15 | 44 | 6-18-20 6-20-18 20-6-18 | 6-16-22 6-22-16 16-6-22 | 8-18-18 18-8-18 | |
| 20 | 46 | 10-16-20 16-20-10 16-10-20 | 6-18-22 18-22-6 18-6-22 | 8-16-22 16-22-8 16-8-22 | |
| | | 8-18-20 18-20-8 18-8-20 | 6-16-24 16-24-6 16-6-24 | 6-20-20 20-6-20 | 18-10-18 18-18-10 |
| 25 | 48 | 8-18-22 18-22-8 18-8-22 | 8-16-24 16-24-8 16-8-24 | 10-18-20 18-20-10 18-10-20 | |
| 30 | | 6-18-24 18-24-6 18-6-24 | 10-16-22 16-22-10 16-10-22 | 6-20-22 20-22-6 20-6-22 | |
| 35 | 50 | 8-20-22 20-22-8 20-8-22 | 18-18-22 18-22-10 18-10-22 | 6-22-22 22-6-22 | |
| 40 | | 10-16-24 16-24-10 16-10-24 | 6-20-24 20-24-6 20-6-24 | 8-18-24 18-24-8 18-8-24 | |
| | 52 | 10-20-22 20-22-10 20-10-22 | 10-18-24 18-24-10 18-10-24 | 8-22-22 22-8-22 | |
| 45 | | 6-22-24 22-24-6 22-6-24 | 8-20-24 20-24-8 20-10-24 | | |

50 Mono-long chain triglycerides according to the present invention are preferred over di-long chain triglycerides in some food applications, for example in cooking oils. Molecular distillation can separate MMLMLM from LLM/LML-type triglycerides, and can shift the composition in carbon number concentration, but it cannot fractionate the triglycerides according to their carbon numbers. Because the composition greatly affects the melting point of triglycerides, fractionation by molecular distillation is an important tool.

55 Non-solvent or solvent crystal fractionation can also fractionate LMM/MLM-type triglycerides from the higher melting LLM/LML triglycerides. The behenic MCT's fractionate without a solvent at about 70°F (21°C), while the stearic or stearic:behenic MCT's fractionate at about 60°F (16°C). Crystallization and filtration are usually repeated two or three times.

Uses of Reduced Calorie Fats

The reduced calorie fats of the present invention can be used as a partial or total replacement for normal triglyceride fat in any fat-containing food composition comprising fat and nonfat ingredients to provide reduced calorie benefits. In order to obtain a significant reduction in calories, it is necessary that at least about 50% of the total fat in the food composition, or at least about 20% of the caloric value of the food, comprise the reduced calorie fat. On the other hand, very low calorie and thus highly desirable food compositions of the invention are obtained when the total fat comprises up to 100% of the reduced calorie fat of this invention, and up to about 50% of the calories.

The present reduced calorie fats are useful in a wide variety of food and beverage products. For example, the fat materials can be used in the production of baked goods in any form, such as mixed, shelf-stable baked goods, and frozen baked goods. Possible applications include, but are not limited to, cakes, brownies, muffins, bar cookies, wafers, biscuits, pastries, pies, pie crusts, and cookies, including sandwich cookies and chocolate chip cookies, particularly the storage-stable dual-textured cookies described in U.S. Patent 4,455,333 of Hong & Brabbs. The baked goods can contain fruit, cream, or other fillings. Other baked good uses include breads and rolls, crackers, pretzels, pancakes, waffles, ice cream cones and cups, yeast-raised baked goods, pizzas and pizza crusts, baked farinaceous snack foods, and other baked salted snacks.

In addition to their uses in baked goods, the reduced calorie fats can be used alone or in combination with other regular calorie fats and oils to make shortening and oil products. Suitable sources of regular fats and oils include, but are not limited to: 1) vegetable fats and oils such as soybean, corn, sunflower, rapeseed, low erucic acid rapeseed, canola, cottonseed, olive, safflower, and sesame seed, 2) meat fats such as tallow or lard; 3) marine oils; 4) nut fats and oils such as coconut, palm, palm kernel, or peanut; 5) milkfat; 6) cocoa butter and cocoa butter substitutes such as shea, or illipe butter; and 7) synthetic fats. Shortening and oil products include, but are not limited to, shortenings, margarines, spreads, butter blends, lards, [cooking and frying oils,] salad oils, popcorn oils, salad dressings, mayonnaise, and other edible oils.

The present reduced calorie fats can also be fortified with vitamins and minerals, particularly the fat-soluble vitamins. U.S. Patent 4,034,083 of Mattson (incorporated by reference herein) discloses polyol fatty acid polyesters fortified with fat-soluble vitamins. The fat-soluble vitamins include vitamin A, vitamin D, vitamin E, and vitamin K. Vitamin A is a fat-soluble alcohol of the formula $C_{20}H_{29}OH$. Natural vitamin A is usually found esterified with a fatty acid, metabolically active forms of vitamin A also include the corresponding aldehyde and acid. Vitamin D is a fat-soluble vitamin well known for use in the treatment and prevention of rickets and other skeletal disorders. "Vitamin D" comprises sterols, and there are at least 11 sterols with vitamin D-type activity. Vitamin E (tocopherol) is a third fat-soluble vitamin which can be used in the present invention. Four different tocopherols have been identified (alpha, beta, gamma and delta), all of which are oily, yellow liquids, insoluble in water but soluble in fats and oils. Vitamin K exists in at least three forms, all belonging to the group of chemical compounds known as quinones. The naturally occurring fat-soluble vitamins are K_1 (phyloquinone), K_2 (menaquinone), and K_3 (menadione). The amount of the fat-soluble vitamins employed herein to fortify the present reduced calorie fat materials can vary. If desired, the reduced calorie fats can be fortified with a recommended daily allowance (RDA), or increment or multiple of an RDA, of any of the fat-soluble vitamins or combinations thereof.

Vitamins that are nonsoluble in fat can similarly be included in the present reduced calorie fats. Among these vitamins are the vitamin B complex vitamins, vitamin C, vitamin G, vitamin H, and vitamin P. The minerals include the wide variety of minerals known to be useful in the diet, such as calcium, magnesium, and zinc. Any combination of vitamins and minerals can be used in the present reduced calorie fat.

The present reduced calorie fats are particularly useful in combination with particular classes of food and beverage ingredients. For example, an extra calorie reduction benefit is achieved when the fat is used with noncaloric or reduced calorie sweeteners alone or in combination with bulking agents. Noncaloric or reduced calorie sweeteners include, but are not limited to, aspartame; saccharin; alitame, thaumatin; dihydrochalcones; cyclamates; steviolosides; glycyrrhizins, synthetic alkoxy aromatics, such as Dulcin and P-4000; sucroloose; suosan; miraculin; monellin; sorbitol, xylitol; talin; cyclohexylsulfamates; substituted imidazolines; synthetic sulfamic acids such as acesulfame, acesulfam-K and n-substituted sulfamic acids; oximes such as perillartine; rebaudioside-A; peptides such as aspartyl malonates and succinilic acids; dipeptides; amino acid based sweeteners such as gem-diaminoalkanes, meta-aminobenzoic acid, L-aminodicarboxylic acid alkanes, and amides of certain alphaaminodicarboxylic acids and gem-diamines; and 3-hydroxy-4-alkyloxyphenyl aliphatic carboxylates or heterocyclic aromatic carboxylates.

Bulking or bodying agents are useful in combination with the reduced calorie fats in many food compositions. The bulking agents can be nondigestible carbohydrates, for example, polydextrose and

cellulose or cellulose derivatives, such as carboxymethylcellulose, carboxyethylcellulose, hydroxypropylcellulose, methylcellulose and microcrystalline cellulose. Other suitable bulking agents include gums (hydrocolloids), starches, dextrins, fermented whey, tofu, maltodextrins, polyols, including sugar alcohols, e.g. sorbitol and mannitol, and carbohydrates, e.g. lactose.

5 Similarly, food and beverage compositions can be made that combine the present reduced calorie fats with dietary fibers to achieve the combined benefits of each. By "dietary fiber" is meant complex carbohydrates resistant to digestion by mammalian enzymes, such as the carbohydrates found in plant cell walls and seaweed, and those produced by microbial fermentation. Examples of these complex carbohydrates are brans, celluloses, hemicelluloses, pectins, gums and mucilages, seaweed extract, and biosyn-
10 thetic gums. Sources of the cellulosic fiber include vegetables, fruits, seeds, cereals, and man-made fibers (for example, by bacterial synthesis). Commercial fibers such as purified plant cellulose, or cellulose flour, can also be used. Naturally occurring fibers include fiber from whole citrus peel, citrus albedo, sugar beets, citrus pulp and vesicle solids, apples, apricots, and watermelon rinds.

These dietary fibers may be in a crude or purified form. The dietary fiber used may be of a single type
15 (e.g. cellulose), a composite dietary fiber (e.g. citrus albedo fiber containing cellulose and pectin), or some combination of fibers (e.g. cellulose and a gum). The fibers can be processed by methods known to the art.

The reduced calorie fats can also contain minor amounts of optional flavorings, emulsifiers, anti-spattering agents, anti-sticking agents, anti-oxidants, or the like.

Of course, judgment should be exercised to make use of appropriate reduced calorie fats and
20 combinations of these fats compositions with other food ingredients. For example, a combination of sweetener and fat would not be used where the specific benefits of the two are not desired. The fat and fat ingredient combinations are used where appropriate, and in the proper amounts.

Many benefits are obtained from the use of the present reduced calorie fats in food and beverage compositions, either when used alone or in combination with the ingredients discussed above. A primary
25 benefit is the calorie reduction achieved when the fat is used as a total or partial fat replacement. This calorie reduction can be increased by using combinations of the present fat compositions with reduced calorie sweeteners, bulking agents, or other reduced calorie or noncaloric fats. Another benefit which follows from this use is a decrease in the total amount of fats in the diet. Foods or beverages made with the reduced calorie fats instead of triglyceride fats will also contain less cholesterol, and the ingestion of these
30 foods can lead to reduced serum cholesterol and thus reduced risk of heart disease.

A related benefit is that the use of the reduced calorie fats allows the production of foods and beverages that are stable in terms of shelf stability and penetration stability. Compositions made with the reduced calorie fats have acceptable organoleptic properties, particularly taste and texture.

Dietary foods can be made with the reduced calorie fats to meet special dietary needs, for example, of
35 persons who are obese, diabetic, or hypercholesterolemic. The reduced calorie fat can be a major part of a low-fat, low-calorie, low-cholesterol diet, and they can be used alone or in combination with drug therapy or other therapy. Combinations of food or beverage products made with the reduced calorie fat can be used as part of a total dietary management regimen, based on one or more of these products, containing the reduced calorie fat alone or in combination with one or more of the above-mentioned ingredients, to provide
40 one or more of the above-mentioned benefits.

This discussion of the reduced calorie fats uses, combinations, and benefits, is not intended to be limiting or all-inclusive. It is contemplated that other similar uses and benefits can be found that will fall within the spirit and scope of this invention.

45

Method of Preparation

The triglycerides used in the reduced calorie fats of the present invention can be prepared by a wide variety of techniques such as:

- 50 (a) random rearrangement of long chain triglycerides (e.g. tristearin or tribehenin) and medium chain triglycerides;
(b) esterification of glycerol with a blend of the corresponding fatty acids;
(c) transesterification of a blend of medium and long chain fatty acid methyl esters with glycerol; and
(d) transesterification of long chain fatty acid glycerol esters (e.g., glyceryl behenate) with medium
55 chain triglycerides.

Random rearrangement of triglycerides is well-known in the art, as is the esterification of glycerol with fatty acids. For discussions on these subjects, see Hamilton et al., Fats and Oils: Chemistry and

Technology, pp. 93-96, Applied Science Publishers Ltd., London (1980), and Swern, Bailey's Industrial Oil and Fat Products, 3d ed., pp. 941-943 and 958-965 (1964), both disclosures incorporated by reference herein. Transesterification is also discussed generally in Bailey's at pp. 958-963.

5 Fatty acids per se or naturally occurring fats and oils can serve as sources of fatty acids for preparing the reduced calorie triglycerides. For example, hydrogenated soybean oil and hydrogenated high erucic acid rapeseed oil are good sources of stearic and behenic acid, respectively. Odd chain length long chain saturated fatty acids can be found in ceratin marine oils. Medium chain saturated fatty acids can be obtained from coconut, palm kernel, or babassu oils. They can also be obtained from commercial medium chain triglycerides, such as the Captex 300 brand sold by Capital City Products of Columbus, Ohio.

10 The tribehenin, useful for making the present triglycerides, can be made from behenic acid or from fractionated methyl behenate by esterification of the acid, or by transesterification of the methyl behenate with glycerol. More importantly, blends of behenic acid and medium chain fatty acids can be esterified with glycerol. Other long chain fatty acids (C₁₈, C₂₀, etc.) can be part of the process. Similarly, methyl ester blends can also be interesterified with glycerol.

15 The present reduced calorie fats are generally made by blending the above-described triglycerides with additional fat or oil ingredients. However, the invention is not limited by the method of preparation; other methods known to the art for making fats or oils can also be used. The fats can be refined, bleached, deodorized, or processed in other ways not inconsistent with the purposes of the invention.

20 The reduced calorie fats can be modified to satisfy specific product performance requirements by additional fractionation. Solvent and non-solvent crystal fractionation or fractional distillation methods (e.g. molecular distillation as described above) can be applied to optimize performance. Standard fractionation methods are discussed in Applewhite, Bailey's Industrial Oil and Fat Products, Vol. 3, 4th ed. (1985), pp. 1-39. John Wiley & Sons, New York, incorporated by reference herein.

25 Fractional distillation of the present reduced calorie fats is not limited to molecular distillation, but can also include conventional distillation (continuous or batch). After synthesis of the fats, it is common to use a conventional batch distillation technique to remove most of the excess medium chain triglycerides, and then continue with molecular distillation. The vacuum requirements are not as strict, and the temperature used can be higher in conventional distillation versus molecular distillation. The conventional distillation temperature is generally between 405° F (207° C) and 480° F (249° C). The absolute pressure is less than 8 mm Hg, more preferably less than 2 mm Hg. The distillation is aided by sparging with steam, nitrogen or other inert gas (e.g., CO₂). The distillation is carried out to remove part of the excess MCT, most of the excess MCT, or to distill also the mono-long chain (MLM and LMM) components.

30 Crystal fractionation of the fats can be carried out with and without solvents, with and without agitation. The crystal fractionation can be repeated several times. Crystal fractionation is particularly effective to remove high melters. Fractionation of behenic MCT without solvents can be used to remove carbon number 50 and 52 LLM and LML components, which in turn alters the melting profile of the fat.

40 Analytical Methods

A. 1 CNP-HPLC Method

45 The carbon number profile of the triglycerides of the present invention can be measured by high performance liquid chromatography (HPLC). The method also measures the percentages of medium chain triglycerides, mono-long chain and di-long chain triglycerides. A triglyceride sample to be analyzed is injected on a reverse phase liquid chromatograph (LC) equipped with a mass (evaporative light scattering) detector. A linear gradient of increasing methylene chloride in acetonitrile is used to separate all of the triglycerides based on fatty acid chain length. Retention time increases with increasing fatty acid chain length. Thus, medium chain triglycerides are eluted first, followed by mono-long chain and then di-long chain triglycerides.

55 Apparatus

Dispensers

1 mL, American Scientific
#P4952-1, or equivalent,
American Scientific Products,
1430 Waukegan Rd., McGaw
Park, IL 60085

5

10

15

20

25

30

35

40

45

50

55

| | | |
|----|--|--|
| 5 | Pasteur pipets, glass | Fisher #13-678-7A, or equivalent, Fisher Scientific Co., 203 Fisher Bldg., Pittsburgh, PA 15219 |
| | Vials, glass | 2 dram with foil-lined cap |
| 10 | Autosampler vials | 2 mL, Fisher #03-340-SG, Fisher Scientific Co. |
| | Vial caps | PTFE Rubber, Fisher #03-340- 13C, Fisher Scientific Co. |
| 15 | LC columns | 2 Beckman Ultrasphere ODS, 5 um, 0.46 cm i.d. x 25 cm, Beckman Instruments, Inc., 2500-T Harbor Blvd., Fullerton, CA 92634 |
| 20 | LC system | Hewlett-Packard 1090L with Ternary DR5 pump, variable volume injector, autosampler, heated column compartment and column switching valve, 25 Hewlett-Packard Co., Scientific Instruments Div., 1601-T California Ave., Palo Alto, CA 94304 |
| 30 | Mass detector | Applied Chromatography Systems #750/14, Varex Corp., 12221 Parklane Dr., Rockville, MD 20852 |
| 35 | Recorder | Kipp & Zonen #BD40, or equivalent, Kipp & Zonen, Div. of Enraf-Nonius, 390-T Central Ave., Bohemia, NY 11716 |
| 40 | Laboratory Automation System (LAS) | Hewlett-Packard 3357, or equivalent, Hewlett-Packard Co., Scientific Instruments Div. |
| 45 | Filters | Gelman #4451, 0.2 um, or equivalent, Gelman Instrument Co., 605-T S. Wagner Rd., Ann Arbor, MI 48106 |
| 50 | Solvent Clarification kit | Waters #85124, Waters Instruments, Inc., 2411-T 7th St. N.W., Rochester, MN 55901 |
| 55 | Syringe | 5 mL, disposable, Fisher #14-823-200, or equivalent, Fisher Scientific Co. |

Reagents

| | | |
|----|-----------------------|--|
| 5 | Methylene chloride | Burdick and Jackson, UV Grade, American Scientific #300-4L, American Scientific Products |
| 10 | Acetonitrile | Burdick and Jackson, UV Grade, American Scientific #015-4L, American Scientific Products |

Sample Preparation

- 15
1. Weigh 0.1 g of the melted sample into a 2 dram vial.
 2. Dispense 1 mL of methylene chloride into vial and mix thoroughly.
 3. Filter the sample solution through a 0.2 um filter into an autosampler vial.

20

LAS Method and Sequence Preparation

- 25
1. Set up the integration method, referring to the HP-3357 Quick Reference Guide for instructions. The calibration table is shown in Table 2.
 2. Set up a LAS sample sequence for the appropriate number of samples. Refer to the Reference Guide as necessary.

30

35

40

45

50

55

Table 2

| Calibration Table | | | | |
|-------------------|-------|----------|----------|-----------|
| | Time | Factor | Amount | Peak Name |
| 1. | 3.48 | 1.000000 | 1.000000 | C22 |
| 2. | 3.80 | 1.000000 | 1.000000 | C24 |
| 3. | 4.18 | 1.000000 | 1.000000 | C26 |
| 4. | 4.30 | 1.000000 | 1.000000 | C28 |
| 5. | 4.65 | 1.000000 | 1.000000 | C30 |
| 6. | 5.32 | 1.000000 | 1.000000 | C32 |
| 7. | 6.01 | 1.000000 | 1.000000 | C34 |
| 8. | 6.80 | 1.000000 | 1.000000 | C36 |
| 9. | 7.87 | 1.000000 | 1.000000 | C38 |
| 10. | 8.98 | 1.000000 | 1.000000 | C40 |
| 11. | 10.31 | 1.000000 | 1.000000 | C42 |
| 12. | 11.88 | 1.000000 | 1.000000 | C44 |
| 13. | 13.49 | 1.000000 | 1.000000 | C46 |
| 14. | 15.35 | 1.000000 | 1.000000 | C48 |
| 15. | 17.28 | 1.000000 | 1.000000 | C50 |
| 16. | 19.49 | 1.000000 | 1.000000 | C52 |
| 17. | 21.60 | 1.000000 | 1.000000 | C54 |
| 18. | 23.87 | 1.000000 | 1.000000 | C56 |
| 19. | 26.18 | 1.000000 | 1.000000 | C58 |
| 20. | 28.50 | 1.000000 | 1.000000 | C60 |
| 21. | 30.77 | 1.000000 | 1.000000 | C62 |
| 22. | 33.03 | 1.000000 | 1.000000 | C64 |
| 23. | 35.24 | 1.000000 | 1.000000 | C66 |

LC OperationA. Start-up

1. Turn on power for the HP1090.
2. Filter all solvents with filtration apparatus.
3. Fill reservoirs with filtered solvent; reservoir A contains acetonitrile and reservoir B contains methylene chloride. Open helium toggle valve on back of LC and degas solvents for at least 5-10 minutes. Close helium toggle valve.
4. Set the mass detector to the following settings:
 Attenuation: 2
 Photomultiplier: 2
 Time Constant: 5
 Evaporator Setting: 50
 Nitrogen: 12 psi
5. Set up the mobile phase gradient method in Table 3 on the HP1090 as necessary. Refer to HP1090 Operator's Handbook for programming directions. Once the method is programmed, it will remain in the memory until it is erased, even with power off or instrument unplugged.

Table 3Mobile Phase Gradient Program

METHOD 1
 TMCT
 5 SDS CONFIG A = 1 B = 1 C = 0
 FLOW = 2
 %B=35 C = 0
 OVEN = 40 INJ VOL = 10 SLOWDOWN = 5
 MAX PRESS = 300
 10 MIN PRESSS = 0
 STOP TIME = 40.1
 POST TIME 5
 COLUMN SWITCH = 0
 E = 0 0 0 0
 15 AT 0 E4 = 1
 AT 0 %B = 35 %C = 0
 AT .1 E4 = 0
 AT 40 %B = 55 %C = 0

20

B. Autosampler Operation

1. Place the filled autosampler vials in autosampler holders starting with space "0". Autosampler starts numbering with "0" and the LAS starts numbering with "1", thus the sequence numbers are shifted by one.
- 25 2. Program and start the autosampler for number of injections, refer to handbook.

Reference Standards

30

A reference standard is used to insure proper LC/detector operation and to verify the identification of the triglyceride peaks. Typically, a well-characterized material is used. When such material is not available, a commercial material such as Nu Chek Prep 50A and 51A can be substituted (Nu Chek Prep, Inc., P.O. Box 172, Elysian, MN 56028). The reference standard is analyzed each day prior to sample analyses.

35

Results

1. As each sample is analyzed, the LAS will generate a report according to the instructions of the integration method (Table 2). The report lists peak number, retention time, and area percent for a given carbon number of the triglyceride sample.
- 40 2. Since retention times of peaks will shift as a function of column usage, verify the proper identification of the reference standards peaks. If peaks are mislabelled, modify the retention time table of the integration method and reanalyze the sequence to generate the new reports.
- 45 3. A chromatogram is often helpful to understand the data. Use CPLOT to generate a chromatogram.

2. CNP-GC Method

- 50 The carbon number profile (CNP) of triglycerides of the present invention can also be determined by programmed temperature-gas chromatography (GC) using a shirt fused silica column coated with methyl silicone for analysis and characterization of the composition by molecular weight. The glycerides are separated according to their respective carbon numbers, wherein the carbon number defines the total number of carbon atoms on the combined fatty acid residues. The carbon atoms on the glycerol molecule are not counted. Glycerides with the same carbon number will elute as the same peak. For example, a triglyceride composed of three C₁₆ (palmitic) fatty acid residues will co-elute with triglycerides made up of one C₁₄ (myristic), one C₁₆ and one C₁₈ (stearic) fatty acid residue or with a triglyceride composed of two C₁₄ fatty acid residues and one C₂₀ (arachidic) fatty acid residue.
- 55

Preparation of the fat sample for analysis is as follows: 1.0 ml. of a tricaprln internal standard solution (2 microg. ml.) is pipetted into a vial. The methylene chloride solvent in the standard solution is evaporated using a steam bath under a nitrogen stream. Two drops of the fat sample (20 to 40 microg.) are pipetted into a vial. If the fat sample is solid, it is melted on a steam bath and stirred well to insure a representative sample. 1.0 ml. of bis (trimethylsilyltrifluoroacetamide) (BSTFA) is pipetted into the vial which is then capped. The contents of the vial are shaken vigorously and then placed in a beating block (temperature of 100° C) for about 5 minutes.

For determining the CNP-GC of the prepared fat samples, a Hewlett-Packard 5880A series gas chromatograph equipped with temperature programming and a hydrogen flame ionization detector is used together with a Hewlett-Packard 3351B data system. A 2 m. long, 0.22 mm. diameter fused silica capillary column coated with a thin layer of methyl silicone (Chromapak CP-SiL 5) is also used. The column is heated in an oven where temperature can be controlled and increased according to a specified pattern by the temperature programmer. The hydrogen flame ionization detector is attached to the outlet port of the column. The signal generated by the detector is amplified by an electrometer into a working input signal for the data system and recorder. The recorder prints out the gas chromatograph curve and the data system electronically integrates the area under the curve. The following instrument conditions are used with the gas chromatograph:

| | |
|----------------|-------------------------|
| Septum purge | 1 ml./min. |
| Inlet pressure | 5 lbs./in. ² |
| Vent flow | 75 ml./min. |
| Makeup carrier | 30 ml./min. |
| Hydrogen | 30 ml./min. |
| Air | 400 ml./min. |

1.0 microl. of the prepared fat sample is taken by a gas-tight syringe and injected into the sample port of the gas chromatograph. The components in the sample port are warmed up to a temperature of 365° C and swept by a helium carrier gas to push the components into the column. The column temperature is initially set at 175° C and held at this temperature for 0.5 min. The column is then heated up to a final temperature of 355° C at a rate of 25° C/min. The column is maintained at the final temperature of 355° C for an additional 2 min.

The chromatographic peaks generated are then identified and the peak areas measured. Peak identification is accomplished by comparison to known pure glycerides previously programmed into the data system. The peak area as determined by the data system is used to calculate the percentage of glycerides having a particular Carbon Number (C_N) according to the following equation:

$$\% C_N = (\text{Area of } C_N / S) \times 100$$

wherein S = sum of Area of C_N for all peaks generated.

The Area of C_N is based upon the actual response generated by the chromatograph multiplied by a response factor for glycerides of the particular Carbon Number. These response factors are determined by comparing the actual responses of a mixture of pure glycerides of various Carbon Numbers to the known amounts of each glyceride in the mixture. A glyceride generating an actual response greater than its actual amount has a response factor less than 1.0; likewise, a glyceride generating a response less than that of its actual amount has a response factor of greater than 1.0. The mixture of glycerides used (in a methylene chloride solution) is as follows:

| Component | Carbon No. | Amount (mg./ml.) |
|------------------|------------|------------------|
| Palmitic acid | 16 | 0.5 |
| Monopalmitin | 16 | 0.5 |
| Monostearin | 18 | 0.5 |
| Dipalmitin | 32 | 0.5 |
| Palmitostearin | 34 | 0.5 |
| Distearin | 36 | 0.5 |
| Tripalmitin | 48 | 1.5 |
| Dipalmitostearin | 50 | 1.5 |
| Distearopalmitin | 52 | 1.5 |
| Tristearin | 54 | 1.5 |

B. Fatty Acid Composition

Principle

The fatty acid composition of the triglycerides comprising the reduced calorie fat of the present invention is measured by gas chromatography. First, fatty acid ethyl esters of the triglycerides are prepared by any standard method (e.g., by transesterification using sodium ethoxide), and then separated on a capillary column which is coated with DB-WAX stationary phase. The fatty acid ethyl esters are separated by chain length and degree of unsaturation. A split injection is made with flame ionization detection. Quantitation is performed by use of a double internal standard method. This method can separate fatty acid ethyl esters from C6 to C24.

Equipment

Gas Chromatograph

Hewlett-Packard 5890, or equivalent, equipped with a split injector and flame ionization detector, Hewlett-Packard Co., Scientific Instruments Div., 1601-T California Ave., Palo Alto, CA 94304

Autosampler Injector column

Hewlett-Packard 7673A, or equivalent

Column

15 m x 0.25 mm I.D., fused silica capillary column coated with DB-WAX (0.25 micron film thickness), Hewlett-Packard Co., Scientific Instruments Div.

Data System

Hewlett-Packard 3350, 3000-T Hanover St., Palo Alto, CA 94304

Recorder

Kipp & Zonen, BD40, Kipp & Zonen

Reagent

Hexane

Burdick & Jackson, or
equivalent, American
Scientific ProductsReference Standards

Two reference standards are used each day of operation to verify proper operation of this method. 1) A reference mixture of fatty acid methyl esters (FAME) is used to check the operation of the instrument. This reference mixture has the following fatty acid composition: 1% C_{14:0}, 4% C_{16:0}, 3% C_{18:0}, 45% C_{18:1}, 15% C_{18:2}, 3% C_{18:3}, 3% C_{20:0}, 3% C_{22:0}, 20% C_{22:1}, and 3% C_{24:0}. A reference standard of a commercial shortening is used to check the operation of the total system -- ethylation and gas chromatographic analysis. The shortening reference standard has the following fatty acid composition: 0.5% C_{14:0}, 21.4% C_{16:0}, 9.2% C_{18:0}, 40.3% C_{18:1}, 23.0% C_{18:2}, 2.2% C_{18:3}, 0.4% C_{10:0}, 1.3% C_{20:1}, and 0.3% C_{22:0}.

The reference mixture of FAME should be diluted with hexane and then injected into the instrument. A new vial of FAME reference mixture should be opened every day since the highly unsaturated components, C_{18:2} and C_{18:3}, oxidize easily. The shortening reference standard should be ethylated with the samples prior to their analysis by capillary gas chromatography. The results from the reference standards should be compared with the known values and a judgment made concerning the completed analysis. If the results of the reference standards are equal to or within \pm standard deviations of the known values, then the equipment, reagents and operations are performing satisfactorily.

OperationA. Instrumental Set-up

1. Install the column in the gas chromatograph, and set up the instrumental conditions as in Table 4.
2. Set up the data system with the appropriate method to acquire and analyze the data. The retention times may have to be adjusted in the method due to instrument variations. Consult the data system reference manual on how to do this -- HP3350 User's Reference Manual. Unity response factors are used for each component.
3. Obtain the shortening reference standard for analysis with the samples and ethylate it with the samples.

Table 4

| INSTRUMENTAL CONDITIONS | | |
|---------------------------|------------------------------|--|
| 5 | Instrument | Hewlett-Packard 5890 |
| | Column | 15 m x 0.25 mm I.D., coated with DB-WAX, 0.25 u film thickness |
| | Column head pressure | 12.5 psi |
| | Carrier gas | Helium |
| | Injector "A" temperature | 210 °C (410 °F) |
| 10 | Split vent flow | 100 mL/min |
| | Septum purge | 1.5 mL/min |
| Oven temperature profile: | | |
| | Initial temperature | 110 °C (230 °F) |
| 15 | Initial time | 1 min |
| | Rate 1 | 15 °C/min |
| | Final temp 1 | 170 °C (338 °F) |
| | Final time 1 | 0 min |
| | Rate 2 | 6 °C/min |
| 20 | Final temp 2 | 200 °C (392 °F) |
| | Final time 2 | 0 min |
| | Rate 3 | 10 °C/min |
| | Final temp 3 | 220 °C (428 °F) |
| | Final time 3 | 8 min |
| 25 | Detector | FID |
| | Detector temp | 230 °C (446 °F) |
| | Make-up gas | 30 mL/min |
| | Detector H ₂ flow | 30 mL/min |
| 30 | Detector air flow | 300 mL/min |

B. Analysis of Samples - (The samples are analyzed with a double internal standard.)

1. Dilute the reference mixture of FAME with hexane. The methyl esters should be approximately 2% in hexane. Inject one microliter of this solution via the autosampler. The results must meet the criteria in the Reference Standards section.

2. Prepare the triglyceride samples to be analyzed by adding two different internal standards, C₉ and C₂₁ triglycerides. (C₉ and C₂₁ triglycerides are commercial standards consisting of 100% 9-carbon and 21-carbon triglycerides, respectively.) The internal standards are added to the samples at about 10% by weight of the sample. The samples (including the internal standards) are then converted to ethyl esters by any standard method.

3. Set up a sequence in the LAS data system to inject the samples.

4. Activate the autosampler to inject 1.0 microl. of the samples in the sequence. The gas chromatograph will automatically begin its temperature program and the data system will collect and analyze the data for the sequence.

5. The data is analyzed with the two internal standard procedure. The absolute amount (mg of esters per gram of sample) of the C₆ through C₁₈ components is calculated from the C₉ internal standard. The absolute amount of the C₁₈, C₂₀, C₂₂ and C₂₄ components is calculated from the C₂₁ internal standard. Weight percentages of fatty acids are calculated from these amounts.

The following examples are intended only to further illustrate the invention and are not intended to limit the scope of the invention which is defined by the claims.

Example 1

A reduced calorie fat according to the present invention is made by random rearrangement of tribehenin

and commercial grade medium chain triglycerides.

The tribehenin is first synthesized by reacting glycerol with a blend of methyl esters having the following fatty acid composition by weight: 7.5% C_{18:0}, 7.4% C_{20:0}, 82.4% C_{22:0}, and 2.7% C_{24:0}. 88.6 lbs. of the methyl ester blend is reacted for 6 hours with 7.2 lbs. of glycerol along with 142 g. of sodium methoxide catalyst in a stainless steel reactor, under a vacuum of 10 mm Hg absolute pressure, and using mechanical agitation, refluxing and nitrogen sparging. During the reaction the temperature gradually rises from 120 °C (248 °F) to 160 °C (320 °F). At the end of 6 hours the reaction mixture is cooled to 120 °C (248 °F) and the catalyst is neutralized with 181 g. of 75% phosphoric acid. The reaction mixture is filtered to remove the sodium phosphate formed. To remove the residual methyl esters, 72 lbs. of the filtered fat is stripped for 4 hours at 240 °C (464 °F) to 280 °C (536 °F) using nitrogen sparging, mechanical agitation and a vacuum of 8-20 mm Hg. absolute pressure, and then stripped for 1 hour using water sparging under the same conditions. The carbon number profile (by HPLC) of the product tribehenin is: 1.3% C₅₈, 2.9% C₆₀, 19.2% C₆₂, 18.4% C₆₄, 48.9% C₆₆, 4.4% C₆₈ and 4.9% others.

24.8 lbs. of the tribehenin undergoes random rearrangement by reaction with 57.9 lbs. of commercial grade medium chain triglycerides in a stainless steel vessel with agitation, using 0.2% sodium methoxide as the catalyst. The medium chain triglycerides contain approximately 68% caprylic acid, 30.5% capric acid, less than 1% caproic acid, and 0.5% lauric acid. The reaction takes place under the following conditions:

| Time | Temperature | Reaction |
|----------------|----------------|---|
| 0 | 190 °F (88 °C) | The reactants are added to the vessel along with 30 g. of the catalyst. |
| 20 min. | 196 °F (91 °C) | 43 more grams of catalyst are added. |
| 40 min. | 172 °F (78 °C) | 15 more grams of catalyst are added. |
| 55 min. | 172 °F (78 °C) | The catalyst is neutralized by adding 118 g. of 75% phosphoric acid. |
| 1 hr., 23 min. | 172 °F (78 °C) | About 1.4 lbs. of activated carbon and about 1.4 lbs. of bleaching earth are added to the reaction mixture. |
| 2 hr., 58 min. | 172 °F (78 °C) | The reaction mixture is filtered through a plate and frame filter using about 400 g. of filter aid (i.e. Kiesel Guhr) |

Seventy-four lbs. of product filtrate is obtained. The carbon number profile of the product as measured by HPLC is shown in Table 5 below as Behenic MCT, Batch 1.

Table 5

Carbon Number Profile and Fatty Acid CompositionStearic-Behenic MCT:

| | | | | | |
|----|------------|----------------|----------------|----------------|----------------|
| 5 | <u>CNP</u> | <u>Batch 1</u> | <u>Batch 2</u> | <u>Batch 3</u> | <u>Batch 4</u> |
| | 24 | 2.4% | | | |
| | 26 | 9.8 | | | |
| 10 | 30 | 6.1 | | | |
| | 32 | 9.4 | 1.2% | | 2.0% |
| | 34 | 25.8 | 17.2 | 3.9% | 19.6 |
| 15 | 36 | 21.8 | 27.3 | 16.9 | 26.9 |
| | 38 | 16.3 | 33.9 | 39.0 | 30.6 |
| | 40 | 5.5 | 16.6 | 27.9 | 16.4 |
| | 42 | 0.6 | 2.9 | 6.7 | 2.5 |
| 20 | 44 | | 1.0 | 3.0 | 0.8 |
| | 46 | | | 1.4 | |
| 25 | <u>FAC</u> | <u>Batch 1</u> | <u>Batch 3</u> | <u>Batch 4</u> | |
| | C8 | 34.6% | 29.8% | 29.1% | |
| 30 | C10 | 22.0 | 16.2 | 13.4 | |
| | C12 | 0.4 | 0.1 | 0 | |
| | C14 | 0 | 0 | 0 | |
| | C16 | 2.9 | 1.1 | 1.4 | |
| 35 | C18 | 24.6 | 14.9 | 18.7 | |
| | C18:1 | 0.2 | 0 | 0 | |
| | C18:2 | 0 | 0 | 0 | |
| 40 | C18:3 | 0 | 0 | 0 | |
| | C20 | 3.1 | 5.7 | 5.1 | |
| | C22 | 10.2 | 28.5 | 19.5 | |
| 45 | C24 | 0.2 | 0.5 | 0.3 | |

*By "0%", as used herein, is meant that none was detected by the available analytical method.

Table 5. cont.Behenic MCT:

| | | Batch | Batch | Batch | Batch | Batch | Batch |
|----|-------------|----------|----------|----------|----------|----------|------------|
| | <u>CNP*</u> | <u>1</u> | <u>2</u> | <u>3</u> | <u>4</u> | <u>5</u> | <u>6**</u> |
| 5 | 22 | 12.9 | | | | | |
| | 24 | 17.9 | 0.4 | | | 1.6 | |
| 10 | 26 | 8.6 | 3.6 | | | 4.4 | |
| | 28 | | 5.9 | | | | |
| | 30 | 1.6 | 4.1 | | 0.7 | 2.7 | |
| 15 | 32 | 2.1 | 1.1 | | 0.5 | 1.5 | 0.1 |
| | 34 | 2.5 | 4.6 | 0.8 | 1.5 | 2.5 | 0.3 |
| | 36 | 4.6 | 13.6 | 9.4 | 9.9 | 9.5 | 2.5 |
| 20 | 38 | 20.6 | 35.3 | 37.7 | 39.5 | 35.5 | 29.0 |
| | 40 | 15.2 | 27.4 | 39.9 | 37.1 | 33.7 | 47.2 |
| | 42 | 3.1 | 6.1 | 13.8 | 10.8 | 8.7 | 17.6 |
| | 44 | 0.4 | | 0.5 | | | 0.9 |
| 25 | 48 | 0.9 | | | | | 0.3 |
| | 50 | 1.1 | | | | | 0.4 |
| | 52 | 5.0 | | | | | 0.4 |
| 30 | 54 | 1.6 | | | | | 0.1 |

*Batches 1-5 by HPLC, Batch 6 by GC

**Subjected to multiple path molecular distillation.

followed by nonsolvent crystal fractionation at
76°F (24.4°C)

Table 5. cont.

| | <u>FAC</u> | <u>Batch 5</u> | <u>Batch 6</u> |
|----|------------|----------------|----------------|
| 5 | C6 | 0.9 | 0.2 |
| | C8 | 28.5 | 22.9 |
| | C10 | 19.3 | 23.8 |
| | C12 | 0.3 | 0.4 |
| 10 | C14 | 0 | 0 |
| | C16 | 0.3 | 0.1 |
| | C18 | 0.7 | 0.8 |
| 15 | C18:1 | 0 | 0.1 |
| | C18:2 | 0 | 0 |
| | C18:3 | 0 | 0 |
| 20 | C20 | 2.6 | 3.4 |
| | C22 | 44.0 | 43.4 |
| | C24 | 0.5 | 1.0 |

25

Stearic MCT:

| | <u>CNP</u> | <u>Batch 1</u> | <u>Batch 2</u> |
|----|------------|----------------|----------------|
| 30 | 24 | | 3.3 |
| | 26 | 5.1 | 8.5 |
| | 28 | | 2.1 |
| | 30 | 3.0 | 2.2 |
| 35 | 32 | 10.9 | 11.3 |
| | 34 | 38.9 | 36.2 |
| | 36 | 31.4 | 28.1 |
| 40 | 38 | 6.5 | 5.5 |
| | 40 | 0.3 | 0.3 |
| | 42 | 0.9 | 0.8 |
| 45 | 44 | 1.6 | 1.6 |

50

Example 2

A stearic:behenic MCT according to the present invention is found to contain a mixture of MMM, MLM, LMM, LLM, LML, and LLL triglycerides. This mixture is fractionated by a molecular distillation process to increase the concentration of mono-long chain triglycerides (MLM and LMM), the long chain fatty acid being predominantly stearic and behenic in this case. A 14" centrifugal molecular still is used for the fractionation, set up as follows:

Bell jar pressure: 0.007 mm Hg. abs.

Degasser pump temperature: 52° C (126° F)

Degasser inlet temperature: 100° C (212° F)

Rotor feed temperature: 127° -160° C (261° -320° F)

Rotor residue temperature: 127° -165° C (261° -329° F)

Feed pump rate: 22 lbs./hour

5 Distillation rate: 1.5 lbs./hr.

The triglyceride mixture is passed through the molecular still eight to fifteen times. The distillation temperature is gradually increased after each pass.

Table 6 below illustrates how the composition of the triglyceride changes during the fractionation, by HPLC carbon number profile and fatty acid composition. After each pass through the molecular still, the distillate is saved and passed again through the molecular still. It is seen that by the fifteenth pass through the molecular still, the product obtained has an increased concentration of C₃₈, C₄₀ and C₄₂ triglycerides. Triglycerides with a carbon number of 32 and below, and 48 and above, have been distilled out from the product. Table 1, discussed above, illustrates some of the possible triglycerides represented by the various carbon numbers. By increasing the concentration of C₃₈, C₄₀ and C₄₂ triglycerides, the fractionation process increases the concentration of the mono-long chain triglycerides (MLM and LMM), and reduces the concentration of the medium chain triglycerides (MMM), di-long chain triglycerides (LLM and LML), and tri-long chain triglycerides (LLL).

A behenic MCT and a stearic MCT are fractionated in a manner similar to that described above. The results are illustrated in Tables 7 and 8.

Table 6
Stearic/Behenic MCT

| | CNP | D9* | D10 | D12 | D14 | D16 | D18 | D27 |
|----|-----|------|------|------|------|------|------|------|
| 25 | 24 | 4.2 | 3.5 | 1.2 | 1.3 | | | |
| | 26 | 9.9 | 8.9 | 4.1 | | | | |
| | 30 | 4.9 | 4.6 | 2.5 | 2.7 | 1.0 | 0.5 | |
| 30 | 32 | 6.5 | 6.4 | 4.9 | 5.4 | 3.2 | 2.0 | |
| | 34 | 26.9 | 27.3 | 27.8 | 27.0 | 25.6 | 21.3 | 5.4 |
| 35 | 36 | 20.9 | 21.5 | 24.8 | 24.2 | 26.6 | 26.0 | 16.2 |
| | 38 | 16.4 | 17.0 | 21.5 | 21.0 | 25.2 | 28.0 | 32.6 |
| | 40 | 6.7 | 7.0 | 9.3 | 9.3 | 11.9 | 14.5 | 26.1 |
| 40 | 42 | 1.2 | 1.2 | 1.5 | 1.7 | 2.1 | 2.8 | 7.3 |
| | 44 | | 0.5 | 0.7 | 0.7 | 1.0 | 1.4 | 4.4 |
| | 46 | | | | | 0.4 | 0.6 | 2.4 |
| 45 | 48 | | | | | 0.5 | 0.7 | 3.2 |
| | 50 | | | | | | | 0.9 |
| | 52 | | | | | | | |
| 50 | 54 | 0.5 | | | | | | |

*"D9", "D10" etc. represent samples of distillate fractions obtained after consecutive passes through the molecular still.

Table 6, continued

Stearic/Behenic MCT

| | <u>FAC</u> | <u>D9</u> | <u>D10</u> | <u>D12</u> | <u>D14</u> | <u>D16</u> | <u>D18</u> | <u>D27</u> |
|----|------------|-----------|------------|------------|------------|------------|------------|------------|
| 5 | C6 | 0.8 | 0.8 | 0.8 | 0.8 | 0.7 | 0.6 | 0.4 |
| | C8 | 33.1% | 33.3% | 32.5% | 32.9% | 31.8% | 31.0% | 25.3% |
| | C10 | 22.7 | 21.4 | 17.5 | 17.5 | 15.5 | 15.8 | 18.5 |
| 10 | C12 | 0.5 | 0.4 | 0.3 | 0.3 | 0.2 | 0 | 0.3 |
| | C14 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | C16 | 2.7 | 2.6 | 2.5 | 2.5 | 2.0 | 1.7 | 1.1 |
| 15 | C18 | 22.6 | 22.3 | 24.6 | 25.0 | 24.1 | 22.9 | 15.9 |
| | C18:1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | C18:2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | C18:3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 20 | C20 | 2.9 | 3.0 | 3.6 | 3.6 | 4.0 | 4.3 | 4.5 |
| | C22 | 9.7 | 10.3 | 13.0 | 12.9 | 15.7 | 18.5 | 29.6 |
| | C24 | 0 | 0.2 | 0.2 | 0.2 | 0.3 | 0.4 | 0.8 |
| 25 | | | | | | | | |
| 30 | | | | | | | | |
| 35 | | | | | | | | |
| 40 | | | | | | | | |
| 45 | | | | | | | | |
| 50 | | | | | | | | |
| 55 | | | | | | | | |

Table 7
Behenic MCT

| | | | | | Distillation | |
|----|------------|--------------|-----------|-----------|--------------|---------------------------|
| | <u>CNP</u> | <u>Feed*</u> | <u>D7</u> | <u>D9</u> | <u>D11</u> | <u>D13</u> <u>Residue</u> |
| 5 | 24 | 5.0 | 1.3 | | | |
| 10 | 26 | 7.6 | 6.6 | 2.0 | | |
| | 30 | 2.7 | 5.7 | 2.7 | 0.8 | |
| | 32 | 1.0 | 4.7 | 3.5 | 1.7 | 0.5 |
| 15 | 34 | 1.1 | 4.7 | 5.4 | 2.7 | 1.1 |
| | 36 | 5.2 | 13.5 | 13.7 | 15.0 | 7.4 |
| | 38 | 28.5 | 32.6 | 36.9 | 39.6 | 38.1 3.1 |
| 20 | 40 | 29.8 | 25.0 | 29.0 | 33.3 | 38.5 12.3 |
| | 42 | 9.8 | 5.2 | 6.7 | 9.3 | 12.5 10.5 |
| 25 | 44 | | | | | 0.4 1.8 |
| | 46 | | | | | 0.6 1.6 |
| | 48 | | | | | 2.7 |
| | 50 | 0.9 | | | | 9.4 |
| 30 | 52 | 5.9 | | | | 34.3 |
| | 54 | 2.6 | | | | 20.8 |
| | 56+ | | | | | 4.1 |

35 *Before fractionation.

40

45

50

55

Table 7, continued

| | | <u>Behenic MCT</u> | | | |
|----|------------|--------------------|-----------|------------|------------|
| | <u>FAC</u> | <u>D7</u> | <u>D9</u> | <u>D11</u> | <u>D13</u> |
| 5 | C6 | 1.3 | 1.2 | 1.0 | 0.8 |
| | C8 | 29.1 | 28.9 | 28.1 | 26.5 |
| | C10 | 21.7 | 17.3 | 16.7 | 18.8 |
| 10 | C12 | 0.6 | 0.4 | 0.3 | 0.3 |
| | C14 | 0 | 0 | 0 | 0 |
| | C16 | 0.7 | 0.5 | 0.3 | 0.2 |
| 15 | C18 | 2.2 | 2.0 | 1.6 | 1.2 |
| | C18:1 | 0 | 0 | 0 | 0 |
| | C18:2 | 0 | 0 | 0 | 0 |
| | C18:3 | 0 | 0 | 0 | 0 |
| 20 | C20 | 4.5 | 4.5 | 4.2 | 3.7 |
| | C22 | 40.1 | 42.2 | 43.2 | 43.7 |
| | C24 | 0.6 | 0.7 | 0.8 | 0.9 |
| 25 | | | | | |

Table 8

| | | <u>Stearic MCT</u> | | | | | |
|----|------------|--------------------|------------|------------|------------|------------|------------|
| | <u>CNP</u> | <u>D11</u> | <u>D12</u> | <u>D13</u> | <u>D14</u> | <u>D15</u> | <u>D16</u> |
| 30 | 24 | 3.0 | 1.5 | 1.9 | 1.1 | 0.4 | 0.3 |
| | 26 | 7.5 | 5.4 | 5.8 | 4.5 | 2.9 | 2.0 |
| 35 | 30 | 3.6 | 2.4 | 3.3 | 2.7 | 1.7 | 1.5 |
| | 32 | 11.1 | 10.4 | 11.3 | 10.3 | 9.9 | 8.6 |
| 40 | 34 | 38.9 | 41.9 | 37.4 | 40.5 | 42.3 | 40.5 |
| | 36 | 28.8 | 32.1 | 29.8 | 31.7 | 34.5 | 34.4 |
| | 38 | 4.9 | 4.9 | 6.2 | 5.9 | 6.1 | 7.5 |
| 45 | 40 | 0.7 | | 1.2 | 0.9 | 0.4 | 1.4 |
| | 42 | 1.8 | | 2.7 | 0.9 | 1.9 | 3.4 |
| | 44 | | 1.3 | 0.4 | 2.4 | | |
| 50 | | | | | | | |

Table 8, continued

Stearic MCT

| <u>FAC</u> | <u>D11</u> | <u>D14</u> | <u>D16</u> |
|------------|------------|------------|------------|
| C6 | 1.1 | 1.1 | 1.0 |
| C8 | 32.9 | 33.3 | 32.4 |
| C10 | 18.6 | 17.1 | 16.5 |
| C12 | 0.3 | 0 | 0 |
| C14 | 0 | 0 | 0 |
| C16 | 4.7 | 4.8 | 4.6 |
| C18 | 35.4 | 38.6 | 40.9 |
| C18:1 | 0 | 0 | 0 |
| C18:2 | 0 | 0 | 0 |
| C18:3 | 0 | 0 | 0 |
| C20 | 0.2 | 0.2 | 0.3 |
| C22 | 0 | 0 | 0 |
| C24 | 0 | 0 | 0 |

Example 3

A strawberry-flavored reduced calorie frozen dessert is made by combining the following ingredients:

| <u>Ingredient</u> | <u>%</u> |
|---|----------|
| Fresh strawberries, finely sliced | 36.25 |
| Stearic-behenic MCT, Batch 1 | 3 |
| Stearic-behenic MCT, Batch 2 | 8 |
| Stearic-behenic MCT, Batch 3 | 5 |
| Polyglycerol ester emulsifier | 0.5 |
| Hexaglycerol monopalmitate emulsifier | 0.3 |
| Propylene glycol monoester emulsifier made from hydrogenated palm oil | 0.3 |
| Blended gum system (Kelco's Dariloid 100) | 0.15 |
| Fructose | 10 |
| Aspartame | 0.02 |
| Vanilla extract | 0.4 |
| Cream solids | 2 |
| Natural cream flavor | 0.05 |
| Artificial vanilla flavor | 0.05 |
| Dried cream extract | 1.0 |
| Soybean lecithin | 0.25 |
| Skim milk | 32.73 |

The carbon number profiles and fatty acid compositions of the stearic-behenic MCT'S are shown above in Table 5. Batch 1 contains 18% medium chain triglycerides (MCT'S) by weight, 82% mono-long chain triglycerides (where the long chain is stearic or behenic) and 0% di-long chain triglycerides (stearic and/or behenic), Batch 2 contains 0% MCT'S, 99% mono-long chain and 1% di-long chain, and Batch 3 contains 0% MCT'S, 94% mono-long chain and 6% di-long chain.

The ingredients are mixed together in a modified batch ice cream making machine equipped with a high shear agitator in the center. The mixture is first warm blended at about 125° F (52° C) for about 10 minutes. Then the mixture is cooled to a temperature of about 24° F (-4° C) over a time of about 23 minutes, under agitation. As the mixture is cooled, it is simultaneously emulsified and the fat is crystallized.

5 Lastly, the mixture is stored at -60° F (-51° C) for two hours and then moved to -20° F (-29° C) storage.

The reduced calorie frozen desserts are found to taste much like premium ice cream.

Example 4

A chocolate-flavored reduced calorie frozen dessert is made as described in Example 4 by combining the following ingredients:

| Ingredient | % |
|---|-------|
| Stearic-behenic MCT, Batch 1 | 3 |
| Stearic-behenic MCT, Batch 2 | 8 |
| Stearic-behenic MCT, Batch 3 | 5 |
| Behenic MCT, Batch 2 | 1 |
| Polyglycerol ester emulsifier | 0.5 |
| Hexaglycerol monopalmitate emulsifier | 0.3 |
| Propylene glycol monoester emulsifier made from hydrogenated palm oil | 0.3 |
| Cocoa | 7 |
| Blended gum system (Kelco's Dariloid 100) | 0.15 |
| Bittersweet Chocolate | 2 |
| Fructose | 5 |
| Sugar (sucrose) | 6 |
| Aspartame | 0.02 |
| Vanilla extract | 0.24 |
| Cream solids | 1 |
| Enzyme modified cream | 1.2 |
| Natural cream flavor | 0.05 |
| Artificial vanilla flavor | 0.05 |
| Dried cream extract | 0.3 |
| Soybean lecithin | 0.25 |
| Skim milk | 58.64 |

The carbon number profiles and fatty acid compositions of the stearic-behenic MCT's and the carbon number profile of the behenic MCT are shown in TABLE 5. The behenic MCT contains 12% MCT's by weight, 88% mono-behenate, and 0% di-behenate.

Example 5

A reduced calorie truffle filling for chocolate-type candy is made as follows. First, the following ingredients are combined and then milled to make a chocolate base material:

| Ingredient | % |
|---|------|
| Behenic MCT, Batch 3 | 26 |
| Soybean lecithin | 0.2 |
| Sugar (sucrose) | 35.8 |
| Nonfat milk solids | 10 |
| Spray-dried buttermilk solids | 8 |
| Cocoa powder, natural process, 19-20% fat | 20 |

The carbon number profile of the behenic MCT is shown in Table 5.

The sugar, milk solids, buttermilk solids and cocoa powder are combined in a mixing bowl and then mixed at low speed while slowly adding 60% of the behenic MCT containing all the lecithin. The remaining behenic MCT is then added in the same manner.

These ingredients are then milled in three passes through a four-roll mill to reduce the particle size. The roll pressures are as follows:

| | Top Roll | 2d Roll | 3d Roll | Bottom Roll |
|----------|----------|----------|---------|-------------|
| 1st pass | 250 psig | 200 psig | 0 | 200 psig |
| 2d pass | 250 | 220 | 0 | 220 |
| 3d pass | 270 | 250 | 0 | 240 |

The product after milling comprises the chocolate base material.

This chocolate base material is then blended with additional ingredients to make a truffle filling:

| Ingredient | % |
|------------------------------|------|
| Behenic MCT, Batch 4 | 9.6 |
| Stearic-behenic MCT, Batch 4 | 2.1 |
| Sugar (sucrose) | 5.5 |
| Vanillin | 0.2 |
| Soybean lecithin | 0.14 |
| Chocolate base material | 82.5 |

The carbon number profile of the behenic MCT and the carbon number profile and fatty acid composition of the stearic-behenic MCT are shown in Table 5.

The filling is prepared by mixing the ingredients at low speed for about 3 minutes, at a temperature of about 120° F (49° C). The mixture is then molded and cooled at 50° F (10° C) for 30 minutes. The truffle filling is coated with a standard milk chocolate to make a good-tasting reduced calorie chocolate-type candy. Alternatively, the truffle filling can be used as a filling in cakes, pies or truffle desserts.

Example 6

A "chocolate" coating for ice cream or other frozen desserts is made from 52 g chocolate liquor, 84 g cocoa (19% fat, natural), 122 g sucrose with very fine particles, 44 g behenic MCT batch 4, 80 g stearic-behenic MCT batch 2, and 0.3 g lecithin (Centracap).

Example 7

A reduced calorie chocolate-type confectionery is made from the following ingredients.

| Ingredient | Grams |
|---------------------|-------|
| Behenic MCT Batch 6 | 226 |
| Soybean Lecithin | 1 |
| Nonfat Milk Solids | 383 |
| Sugar (sucrose) | 1196 |
| Cocoa Powder | 328 |
| Vanilla Flavor | 8 |

The sugar, nonfat milk solids, cocoa powder and vanilla flavor are combined in a mixing bowl with a melted blend of behenic MCT and soybean lecithin. This mixture is then blended thoroughly in a Hobart mixer. The blend is passed through a 4-roll mill having the pressure settings indicated below to reduce the particle size of the sugar:

| Roll | Pressure (psig) |
|--------|-----------------|
| Top | 220 |
| 2nd | 225 |
| 3rd | 0 |
| bottom | 220 |

An additional 205 g. of the behenic MCT (Batch 6) is added to 2264 g. of this milled mixture. This mixture is again passed through the 4-roll mill using the above roll pressure settings to provide a milled confectionery base.

850 g. of this milled confectionery base is blended with the following additional ingredients at 125° F (51.7° C):

| Ingredient | Grams |
|------------------------|-------|
| Soybean Lecithin | 0.5 |
| Behenic MCT (Batch 6) | 90 |
| Dehydrated Sweet Cream | 8 |
| Butter Flavor | 1.6 |
| Chocolate Liquor | 45 |

The temperature of the above blend is reduced to 87° F (30.6° C) and then 100 g. of milled confectionery base that has been aged at room temperature is added. The mixing is continued at approximately 87° F (30.6° C) for another 16 hours using a Kitchen Aid mixer set at slow speed. This conched mixture is then poured into chocolate molds and cooled to 56° F (13.3° C) for 45 min. The cooled molds are placed in an isolated box and then moved to a 60° F (15.6° C) temperature room for 24 hours. Tempering is continued in a 70° F temperature room for another 48 hours.

Claims

1. A reduced calorie fat characterized in that it has an at least 10% reduction in calories relative to corn oil and and further characterized in that it comprises at least 15% by weight reduced calorie triglycerides selected from the group consisting of MML, MLM, LLM, and LML triglycerides, and mixtures thereof and at least about % by weight of a mixture of MML and MLM triglycerides; wherein M = fatty acids selected from the group consisting of C₆ to C₁₀ saturated fatty acids, and mixtures thereof, and L = fatty acids selected from the group consisting of C₁₇ to C₂₆ saturated fatty acids, and mixtures thereof; and wherein the fat has the following fatty acid composition by weight percent:

- (a) from 15 to 70% C₆ to C₁₀ saturated fatty acids;
- (b) from 10 to 70% C₁₇ to C₂₆ saturated fatty acids;
- (c) not more than 10% fatty acids selected from the group consisting of C_{12:0} and C_{14:0}, and mixtures thereof;

(d) not more than 20% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, C_{18:3}, and mixtures thereof; and

(e) not more than 4% C_{18:2} fatty acids.

5 2. A fat-containing food composition having fat ingredients and nonfat ingredients characterized in that from 50 to 100% of the total fat by weight comprises a reduced calorie fat, said reduced calorie fat having an at least 10% reduction in calories relative to corn oil and comprising at least 15% by weight reduced calorie triglycerides selected from the group consisting of MML, MLM, LLM, and LML triglycerides, and mixtures thereof; wherein M = fatty acids selected from the group consisting of C₆ to C₁₀ saturated fatty acids, and mixtures thereof, and L = fatty acids selected from the group consisting of C₁₇ to C₂₆ saturated fatty acids, and mixtures thereof; and wherein the fat has the following fatty acid composition by weight percent:

(a) from 15 to 70% C₆ to C₁₀ saturated fatty acids;

(b) from 10 to 70% C₁₇ to C₂₆ saturated fatty acids;

15 (c) not more than 10% fatty acids selected from the group consisting of C_{12:0} and C_{14:0}, and mixtures thereof;

(d) not more than 20% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, C_{18:3}, and mixtures thereof; and

(e) not more than 4% C_{18:2} fatty acids.

20

3. The fat or food composition of any of Claims 1 to 2 characterized in that it comprises at least 30% by weight of said reduced calorie triglycerides, preferably at least 50% by weight of said reduced calorie triglycerides, and most preferably at least 70% by weight of said reduced calorie triglycerides.

4. The fat or food composition of any of Claims 1 to 3 characterized in that the fat comprises at least 25 35% by weight of said mixture of MML and MLM triglycerides and no more than 65% by weight combined LLM and LML triglycerides.

5. The fat or food composition of any of Claims 1 to 4 characterized in that the fat comprises at least about 93% by weight of a mixture of MML and MLM triglycerides, no more than about 7% by weight combined LLM and LML triglycerides, no more than about 1% by weight MMM triglycerides and no more than about 1% by weight LLL triglycerides.

6. The fat or food composition of any of Claims 1 to 5 characterized in that the fatty acid composition comprises not more than 5% C_{6:0} fatty acid, and not more than 7% saturated C₂₄ to C₂₆ fatty acids.

7. The fat or food composition of any of Claims 1 to 6 characterized in that the fatty acid composition comprises from 30 to 55% C₈ to C₁₀ saturated fatty acids and from 30 to 55% C₁₈ to C₂₂ saturated fatty acids.

8. The fat or food composition any of Claims 1 to 7 characterized in that the fatty acid composition comprises not more than 10% C_{16:0} fatty acid, not more than 3% fatty acids selected from the group consisting of C_{12:0} and C_{14:0} fatty acids, and mixtures thereof, and not more than 6% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, and C_{18:3} fatty acids, and mixtures thereof.

9. The fat or composition of any of Claims 1 to 7 characterized in that the fat has the following carbon number profile (CNP):

45

50

55

| CNP | % |
|--------------|-------|
| 30 or lower | < 0.4 |
| 34 | < 0.3 |
| 36 | < 3.5 |
| 38 | 22-42 |
| 40 | 32-55 |
| 42 | 5-30 |
| 44 | <1.5 |
| 46 | <1 |
| 48 | <0.8 |
| 50 | <0.6 |
| 52 | <0.4 |
| 54 or higher | <0.9 |

10. The fat or food composition of any of Claims 1 to 8 characterized in that said reduced calorie triglycerides are stearic MCT'S having a carbon number profile of at least about 55% C34 to C38, and have a fatty acid composition comprising at least 40% C₈ to C₁₀ saturated fatty acids and from 35 to 50% C_{18:0} fatty acid.

5 11. The fat or food composition of any of Claims 1 to 10 characterized in that the fat has an at least 30% reduction in calories relative to corn oil, preferably between 20 and 50% reduction in calories relative to corn oil.

12. The food composition of any of Claims 2 to 11 characterized in that it is a margarine, shortening, baked good, frozen dessert, confectionery, or chocolate-type product.

10

15

20

25

30

35

40

45

50

55

12

EUROPEAN PATENT APPLICATION

21 Application number: 88202854.1

61 Int. Cl.⁵: **A23D 3/00, A23D 5/00,**
A23G 1/00, A23G 9/02

22 Date of filing: 13.12.88

30 Priority: 15.12.87 US 132400

43 Date of publication of application:
 28.06.89 Bulletin 89/26

84 Designated Contracting States:
 AT BE CH DE FR GB GR IT LI LU NL SE

88 Date of deferred publication of the search report:
 24.10.90 Bulletin 90/43

71 Applicant: **THE PROCTER & GAMBLE**
COMPANY
 One Procter & Gamble Plaza
 Cincinnati Ohio 45202(US)

72 Inventor: **Seiden, Paul**
 2890 Grandin Road
 Cincinnati Ohio 45208(US)

74 Representative: **Suslic, Lydia et al**
 Procter & Gamble European Technical
 Center N.V. Temselaan 100
 B-1820 Strombeek-Bever(BE)

54 **Reduced calorie fats made from triglycerides containing medium and long chain fatty acids.**

57 A reduced calorie fat comprising at least about 15% by weight triglycerides selected from the group consisting of MML, MLM, LLM, and LML triglycerides, and mixtures thereof; wherein M = fatty acids selected from the group consisting of C₆ to C₁₀ saturated fatty acids, and mixtures thereof, and L = fatty acids selected from the group consisting of C₁₇ to C₂₆ saturated fatty acids, and mixtures thereof is disclosed. The fat also has the following fatty acid composition: (a) from about 15% to about 70% C₆ to C₁₀ saturated fatty acids; (b) from about 10% to about 70% C₁₇ to C₂₆ saturated fatty acids; (c) not more than about 10% fatty acids selected from the group consisting of C_{12:0} and C_{14:0}, and mixtures thereof; (d) not more than about 20% fatty acids selected from the group consisting of C_{18:1}, C_{18:2}, C_{18:3}, and mixtures thereof; and (e) not more than 4% C_{18:2} fatty acids.

EP 0 322 027 A3



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

EP 88 20 2854

| DOCUMENTS CONSIDERED TO BE RELEVANT | | | |
|---|---|--|--|
| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (Int. Cl.4) |
| A | DE-A-1 806 159 (ETHYL CORP.) * Claims 1-3; page 4, column 2 - page 6; examples 1-5 * | 1,2 | A 23 D 3/00 A 23 D 5/00 A 23 G 1/00 A 23 G 9/02 |
| A | EP-A-0 074 146 (UNILEVER PLC) * Claims 1,14,15; page 1, line 26 - page 2, line 20; table 2 * | 1,2 | |
| A | GB-A- 816 343 (THOMAS HEDLEY & CO.) * Claim 1; page 1, line 64 - page 2, line 26 * | 1,2 | |
| A | BE-A- 546 643 (THE PROCTER & GAMBLE CO.) * Page 3, column 3 - page 4, column 1 * | 1,2 | |
| D,A | US-A-3 353 964 (P. SEIDEN) * Claim 1; column 3, line 43 - column 4, line 4 * | 1,2 | |
| | | | TECHNICAL FIELDS SEARCHED (Int. Cl.4) |
| | | | A 23 D |
| The present search report has been drawn up for all claims | | | |
| Place of search THE HAGUE | | Date of completion of the search 10-08-1990 | Examiner VUILLAMY V.M.L. |
| CATEGORY OF CITED DOCUMENTS | | | |
| X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document | | T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document | |

